

भारतीय मानक
रबड़ उद्योग के लिए जिंक ऑक्साइड — विशिष्टि
(तीसरा पुनरीक्षण)

Indian Standard
ZINC OXIDE FOR RUBBER INDUSTRY —
SPECIFICATION
(*Third Revision*)

ICS 83.040.20

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FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Rubber and Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council

This standard was first revised in 1973. The second revision of this standard was published in 1993 when classification of zinc oxide was introduced based on the manufacturing process besides the requirements of colour, nitrogen surface area and bulk density.

In this third revision, procedure for determination of zinc oxide has been aligned with corresponding International Standard ISO 9298 : 1995 'Rubber compounding ingredients — Zinc oxide — Test methods'. Determination of residue on sieve has been aligned with International Standard ISO 787-7 : 1981 'General methods of test for pigments and extenders — Part 7: Determination of residue on sieve — Water method — Manual procedure'.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
**ZINC OXIDE FOR RUBBER INDUSTRY —
 SPECIFICATION**
(Third Revision)

1 SCOPE

This standard prescribes the classification, requirements and methods of sampling and tests of zinc oxide for use in rubber industry.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
265 : 1987	Specification for hydrochloric acid (<i>third revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
7086 (Part 1) : 1973	Methods of sampling and test for rubber compounding ingredients, Part 1
12076 : 1986	Precipitated silica for rubber industry

3 CLASSIFICATION

3.0 The type of zinc oxide used in the rubber industry and related to the production process.

3.1 French Process or Indirect Type

When zinc oxide is manufactured by the burning of zinc vapour (produced by boiling zinc metal) it is called French Process or indirect type. It is characterized by a high degree of chemical purity. The particle shape is nodular and the size is 0.2-0.5 micron.

3.2 Secondary Zinc Oxide Type

When zinc oxide is manufactured as a by-product of chemical reaction or results from burning zinc vapour produced from die cast scrap zinc, galvanized zinc dross, it is called as secondary zinc oxide. The particle shape is nodular and the size is 0.2-0.7 micron.

4 REQUIREMENTS

The material shall comply with the requirements given in Table 1 when tested according to the procedures given in col 5 of Table 1.

5 PACKING AND MARKING**5.1 Packing**

The material shall be packed in suitable package as agreed to between the purchaser and the supplier.

5.2 Marking

Each package shall be marked with:

- a) Name and grade of material,
- b) Indication of the source of manufacture,
- c) Net mass of the material,
- d) Month and year of manufacture, and
- e) Lot/Batch number.

5.2.1 BIS Certification Marking

Each package may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 The representative samples of the material shall be drawn as prescribed in IS 7086 (Part 1).

6.2 Number of Tests

Tests for determination of manganese and copper shall be conducted on individual samples. Tests for all other characteristics shall be conducted on the composite sample.

6.3 Criteria for Conformity**6.3.1 For Individual Samples**

The mean and range of the test results for manganese

Table 1 Requirements of Zinc Oxide for Rubber Industry
(Clauses 4 and 7.1)

SI No.	Characteristic	Requirement		Methods of Test, Ref to Cl of IS 7086 (Part 1)/ Annex of This Standard
		French Process Indirect Type	Secondary Type	
(1)	(2)	(3)	(4)	(5)
i)	Colour	White powder	Light yellow	Visual
ii)	Zinc oxide (as ZnO), on dried sample at 105°C, percent by mass, <i>Min</i>	99.5	99.0	A
iii)	Matter insoluble in hydrochloric acid, percent by mass, <i>Max</i>	0.15	0.15	9
iv)	Moisture content, percent by mass, <i>Max</i>	0.25	0.25	7
v)	Sieve residue, percent by mass, <i>Max</i>			
	a) through 45-micron IS sieve	0.15	0.15	B
	b) through 75-micron IS sieve	0.05	0.05	B
vi)	Relative density at 27/27°C	5.55 to 5.68	5.55 to 5.68	4
vii)	Nitrogen surface area m ² /g	3.5 to 5.5	3.5 to 5.5	A-8 of IS 12076
viii)	Lead (as Pb), percent by mass, <i>Max</i>	0.10	0.10	14
ix)	Copper (as Cu), percent by mass, <i>Max</i>	0.001	0.001	12
x)	Manganese (as Mn), percent by mass, <i>Max</i>	0.003	0.003	11
xi)	Bulk density ¹⁾ g/lit	To meet customer requirement		

¹⁾Bulk density of zinc oxide made by American process will be typically in the range of 820 to 950 g/lit and bulk density of zinc oxide made by French Process will be typically between 450 to 550 g/lit.

and copper shall be calculated as follows:

$$\text{Mean } (X) = \frac{\text{The sum of test results}}{\text{No. of the test results}}$$

Range (*R*) = The difference between the maximum and the minimum value of the test results.

The lot shall be deemed to have satisfied the requirements of the specification if,

$$X + 0.6 R < 0.003 \text{ for manganese and} \\ < 0.001 \text{ for copper}$$

6.3.2 For Composite Sample

In respect of all other characteristics, the lot shall be

considered as conforming to the specification if the composite sample satisfies each of these requirements.

7 TEST METHODS

7.1 Tests shall be conducted according to the methods prescribed in Annex A. Reference to relevant clauses of IS 7086 (Part 1) are given in col 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[Clause 7.1, and Table 1, Sl No. (ii)]

DETERMINATION OF ZINC OXIDE CONTENT

A-1 REAGENTS

All reagents shall be of recognized grade and distilled, deionized or distilled/deionized water shall be used for sample preparation and required dilutions.

A-1.1 Nitric Acid — 65 percent (*m/m*)

A-1.2 Hydraulic Acid — 20 percent (*m/m*)

A-1.3 Ammonia Solution — 25 percent (*m/m*).

A-1.4 Hydrogen Peroxide Solution — 3 percent (*m/m*).

A-1.5 Iron (III) Solution

Dissolve 86 g of iron (III) ammonium sulfate in water and dilute to 1 000 cm³.

A-1.6 Ammonium Chloride Solution

Dissolve 250 g of ammonium chloride in water and dilute to 1 000 cm³.

A-1.7 Masking Solution

Dissolve 30 g of ammonium fluoride, 100 g of ammonium thiosulfate and 250 g of ammonium acetate in water and dilute to 1 000 cm³.

A-1.8 Bromothymol Blue Solution

Dissolve 0.1 g of bromothymol blue in 100 cm³ of ethanol.

A-1.9 Xylenol Orange Solution

Dissolve 0.2 g of xylenol orange, tetrasodium salt in 100 cm³ of water.

A-1.10 EDTA, standard volumetric solution, (EDTA) = 0.1 mol/dm³.

Dissolve 37.225 g of ethylenedinitrilotetraacetic acid, disodium salt (Na₂EDTA), in water in a 1 000 cm³ one-mark volumetric flask, dilute to the mark and mix well. Alternatively, commercially available standard solutions may be used.

A-1.11 Zinc Metal, of minimum purity — 99.995 percent (*m/m*).

A-2 APPARATUS

A-2.1 Volumetric Flasks — Class A, of capacity 250 cm³, and 1 000 cm³.

A-2.2 Pipettes — Class A, of capacity 50 cm³, and 1 00 cm³.

A-2.3 Burette — Class A, of capacity 50 cm³.

A-2.4 Balance — of capacity 250 g, weighing to an accuracy of at least ±1 mg.

A-2.5 Heating Device**A-2.6 Acid-Washed, Fluted Filter Paper**

A-2.7 Beakers — of capacity 600 cm³ and 1 000 cm³.

A-2.8 Conical Flasks — of capacity 500 cm³ and 1 000 cm³.

A-3 SAMPLING

Take a representative sample in accordance with IS 7086 (Part 1).

A-4 PROCEDURE

Suspend 20 g of the zinc oxide sample, weighed to ± 0.01 g, in 100 cm³ of water in a 1 000 cm³ beaker (A-2.7) and dissolve carefully with approximately 90 cm³ of nitric acid (A-1.1). When the zinc oxide has dissolved, boil for a short time, cool down the solution and transfer it to a 500 cm³ volumetric flask (A-2.1). Carefully dilute the solution to the mark with water and shake.

Pipette 50 cm³ of this solution into a 250 cm³ volumetric flask (A-2.1). Add 10 cm³ of iron (III) solution (A-1.5). Shake, and then add successively 5 cm³ of hydrogen peroxide solution (A-1.4), 60 cm³ of ammonium chloride solution (A-1.6) and 30 cm³ of ammonia solution (A-1.3).

Shake briefly and cool down. Make up to the mark and filter through a dry folded filter paper (A-2.6) into a dry 500 cm³ conical flask (A-2.8). Pipette 50 cm³ of this solution into a 600 cm³ beaker (A-2.7) and dilute with water to about 300 cm³.

Add four drops of bromothymol blue solution (A-1.8) and neutralize with hydrochloric acid (A-1.2). The colour changes from blue to light yellow. Add 2 drops of hydrochloric acid in excess. After addition of 20 ml of masking solution (A-1.7) and 7 drops of xylenol orange solution (A-1.9), titrate with EDTA solution (A-1.10) until the colour changes from purple-red to orange-yellow.

After further dropwise addition of 0.5 to 1 cm³ of EDTA solution, the colour changes sharply to pale yellowish-green. Let the total volume of EDTA solution added be V_1 .

A-5 STANDARDIZATION PROCEDURE

Dilute concentrated nitric acid (A-1.1) to a concentration of approximately 30 percent (*m/m*).

WARNING— Acid should be added carefully to water.

Then dissolve 20 g of refined zinc (A-1.11), weighed to ± 0.01 g, by heating in a beaker with 40 cm³ of the diluted nitric acid. Allow the solution to cool, transfer to a 1 000 cm³ volumetric flask (A-2.1) and dilute to the mark. Proceed as in A-4, starting at para 2, to obtain the titration volume V_2 .

1 cm³ of 0.1 mol/ dm³ EDTA solution corresponds to 0.006 537 g of zinc or 0.008 138 g of zinc oxide.

A-6 EXPRESSION OF RESULTS

Calculate the total zinc oxide content W_{ZnO} , expressed as a percentage by mass, using the equation

$$W_{\text{ZnO}} = \frac{V_1 \times 100 \times 1.245}{V_2}$$

where

V_1 = volume, in cubic centimetres, of EDTA solution (A-1.10) required for titration of the zinc in the test portion in A-4;

V_2 = volume, in cubic centimetres, of EDTA solution (A-1.10) required for titration of the zinc in the standardization procedure (A-5); and

1.245 = ratio of the relative molecular mass of zinc oxide to the relative atomic mass of zinc.

A-7 INTERFERENCE

Lead and iron are precipitated as hydroxides when the ammonia solution is added. Copper will be masked by ammonium thiosulfate. Aluminium will be masked by ammonium fluoride. Cadmium is also titrated with the EDTA solution, but since the cadmium concentration is usually lower than 0.1 percent (*m/m*) in rubber grade oxides, this error is negligible.

ANNEX B

[Clause 7.1, and Table 1, Sl No. (v)]

DETERMINATION OF RESIDUE ON SIEVE**B-1 APPARATUS**

Ordinary laboratory apparatus

B-1.1 Sieve, of nominal mesh aperture of 45 μm and 75 μm .

B-1.2 Brush, hog bristle, approximate dimensions 5 mm thick, 20 mm wide, 35 mm long.

B-1.3 Sintered Glass Crucible, of porosity grade P 40 (pore size index 16 to 40 μm), or weighing bottle.

B-1.4 Oven, capable of being maintained at $105 \pm 2^\circ\text{C}$.

B-1.5 Balance, accurate to 1 mg or better.

B-1.6 Desiccator, containing an efficient desiccant.

B-1.7 Washbottle, containing the solution used to disperse the test portion.

B-2 SAMPLING

Take a representative sample of the material as per IS 7086 (Part 1).

B-3 PROCEDURE

Carry out the determination in duplicate.

B-3.1 Test Portion

Weigh, to the nearest 0.1 g, into a beaker of suitable capacity, a quantity of the sample such that a sufficient residue on the sieve (3.1) is obtained. Generally, a test portion of 10 to 100 g is necessary, but in the case of products yielding a very low residue on the sieve a larger test portion, up to 1 000 g, should be used.

B-3.2 Preparation of the Dispersion

Disperse the test portion (3.1) in a suitable volume of water (about 300 to 600 ml) containing, if required, a suitable dispersing agent (*see* Note 1). If the product specification advises that mechanical assistance is commonly required to achieve thorough dispersion, a stirrer and stirrer head shall be used, and it is recommended that the rotation of the stirrer should not exceed 500 ± 50 r/min.

NOTES

1 The quantity of the dispersing agent should be between 0.2 and 0.5 percent of the mass of the test portion. The type and quantity of the dispersing agent to be used should be agreed between the parties.

2 It is important that the dispersion of the pigment or extender in the aqueous medium should be thorough and that no flocculation should occur during the determination (B-3.3).

B-3.3 Determination

Pour the dispersion, if necessary in portions, through the sieve (B-1.1). With the aid of the washbottle (B-1.7) filled with the solution used to disperse the test portion, rinse out the beaker and pour all the rinsings through the sieve. Wash the test portion with the same solution until the washings passing through the sieve are clear and free of dispersed material. During the process, the duration of which should not exceed 5 min, care should be taken to avoid the destruction of agglomerates. Finally wash any adherent particles with the brush (B-1.2) into the sieve and wash the residue on the sieve with water to free it from the dispersing agent.

NOTE — In order to avoid false test results, the water should be filtered by a suitable filtering device.

Treat the residue on the sieve according to one of the following alternative procedures:

- a) Wash the residue into the previously heated and weighed sintered glass crucible (B-1.3) and dry in the oven (B-1.4) at $105 \pm 2^\circ\text{C}$ for 1 h. Allow to cool in the desiccator (B-1.6) and weigh to the nearest 1 mg. Repeat the heating for at least 30 min, allow to cool in the desiccator, insert the stopper and again weigh to the nearest 1 mg. Repeat this procedure until two successive weighings differ by not more than 5 mg. Record the lower mass.
- b) Transfer the residue with distilled water into a previously heated and weighed 50 ml beaker. Evaporate the water and dry in the oven at $105 \pm 2^\circ\text{C}$ for 1 h. Continue as described under a) above.
- c) Dry the residue on the sieve in the oven at $105 \pm 2^\circ\text{C}$ for 1 h. Transfer the residue into

the previously heated and weighed weighing bottle (B-1.3) and weigh to the nearest 1 mg. Repeat the heating for at least 30 min, allow to cool in the desiccator, insert the stopper and again weigh to the nearest 1 mg. Repeat this procedure until two successive weighings differ by not more than 5 mg. Record the lower mass.

If the two determinations differ by more than 10 percent of the larger value (unless the difference is less than 5 mg), repeat the procedure.

B-3.4 Examination of the Residue

Inspect the residue for the presence of incompletely dispersed pigment or extender and, if present, repeat the whole procedure using an alternative dispersing agent agreed between the parties.

If the residue contains extraneous matter, report its presence and nature.

B-4 EXPRESSION OF RESULTS

B-4.1 Calculation

Calculate the residue on sieve by the equation

$$R = \frac{100 \times m_1}{m_0}$$

where

R = residue on sieve, expressed as a percentage by mass;

m_0 = mass, in grams, of the test portion; and

m_1 = mass, in grams of residue.

Calculate the mean of two determinations and report the result to two significant figures. If the mean value is below 0.01 percent, report the result as “less than 0.01 percent”.

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